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(54) Title: COATING PROCESSES

(57) Abstract

It is known in curtain coating to use a bottom or accelerating layer to promote wetting of a moving support on to which a liquid material comprising one or more layers is being deposited. Although high coating speeds can be obtained, it is difficult to deliver a low viscosity and relatively low flow rate layer as a bottom layer down the inclined slide of a hopper, without waves and other manifestations of unstable flow. Described herein is an improved process for curtain coating in which a thin, low viscosity bottom layer is used allowing high viscosity liquids to be deposited simultaneously on to a moving support without the problems associated with known curtain coating processes.

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COATING PROCESSES

This invention relates to coating processes and is more particularly concerned with curtain coating processes.

In curtain coating as described in US-A-3867901 and US-A-3508947, one or more liquid layers are applied to a moving support as a freely-falling curtain. Under properly controlled conditions, uniform coatings are achieved in which each layer retains its separate identity. The curtain can be formed using a conventional multilayer slide hopper fitted with a suitable lip. Edging rods may be used to maintain the width of the curtain, and surface active agents are frequently added to the outer layers to improve curtain stability and promote uniform deposition.

In the coating of photographic materials and other products in which one or more liquid layers are applied to a moving support, uniform coatings are obtained only if the operational variables are held within fairly precise limits. These limits define the so-called "coating window".

One important variable which affects the "coating window" is coating speed. For economic reasons, high coating speeds are desirable provided they can be achieved with low waste and without loss of product quality. However, a major problem with high speed coating is that if the speed is too high, the liquid fails to wet the support in a complete and steady manner. When this occurs, air is entrained at the wetting line and causes patchy or uneven deposition of the coating.

Air-entrainment is believed to occur whenever the dynamic contact angle formed between the liquid and the support at the wetting line approaches its limiting

value of 180°. The coating speed at which this happens depends on many factors such as the method of coating and the physical properties of the liquid. The viscosity of the liquid being brought into contact with the support at the wetting line is of particular importance.

In curtain coating as well as in bead coating, the occurrence of air-entrainment can be postponed to higher speeds by reducing the viscosity of the liquid. On the other hand, for aqueous photographic melts and other water or solvent based coating systems, the ability to coat at high viscosities has several advantages such as reduced drying load and improved uniformity.

In multilayer bead coating, a liquid having a relatively low viscosity (typically 3 to 5mPas) is sometimes used to provide a bottom layer for higher viscosity liquids. The bottom layer is added to the higher viscosity liquids out of the bottom slot of the hopper (that is, the slot nearest the bead). Although increased coating speeds are possible, a non-uniform coating is often produced, particularly when the bottom layer is relatively thin, for example, less than about 30mm.

US-A-4569863 discloses a curtain coating process for applying a multiple coating on to a moving support. The process enables a plurality of comparatively high viscosity layers (having viscosities greater than 50mPas) to be applied to the support at coating speeds of 400mmin^{-1} or more. This is achieved by the use of a thin bottom layer (otherwise called an accelerating layer) of lower viscosity. The thickness of this layer is between 2 and 30mm, and its viscosity between 1 and 20mPas. Preferably, the thickness is between 2.5 and 5mm, and the viscosity between 2 and

3mPas. The use of an upper spreading layer is also disclosed.

In US-A-4569863, the bottom or accelerating layer is applied directly to the back of the curtain either by discharging the layer from the bottom slot of a conventional slide hopper, or from a slot which flows on to a separate slide which joins at the hopper lip the main hopper slide on which are flowing the other layer compositions. Hoppers designed for this purpose are sometimes known as V-hoppers.

It should be noted that in US-A-4569863, the freely-falling curtain impinges on to the moving support at an application angle of 0°. (The application angle may be defined as the slope angle of the support at the point of impingement of the freely-falling and substantially vertical curtain measured as a declination from the horizontal in the direction of coating.) Furthermore, the height of the curtain is restricted within the range of 10 to 100mm, preferably 15 to 50mm.

Although the method described in US-A-4569863 can yield high coating speeds, the method suffers from several disadvantages. It is difficult to deliver a low viscosity and relatively low flow rate layer down an inclined hopper slide as a bottom layer without waves and other manifestations of unstable flow. In the arrangement wherein the low viscosity bottom layer is delivered down a separate slide to join the main hopper slide, a long main slide results. This is undesirable since waves and other manifestations of unstable flow on the slide grow very rapidly as slide length is increased, and undesirable restrictions on the relative flow rates and viscosities of the layers on the main slide may result. It is therefore preferred that the total length of the slide of a

multiple slot hopper is kept to a minimum to minimize such restrictions.

The method described in US-A-4569863 may also increase the undesirable tendency of the plane of the curtain to deviate from the vertical, and bend backwards towards the hopper. Also, when the low viscosity layer which wets the support is delivered using a V-hopper, it faces in a downward direction. Thus, the establishment of flow on such a slide can be difficult in practice, and dripping of composition off the slide surface may occur. Furthermore, with this slide orientation, there is a component of gravity normal to the slide surface which is de-stabilizing and promotes the growth of waves on the layer as it travels down the slide.

A low viscosity bottom layer also promotes "puddling" at the point where the freely-falling liquid curtain impinges on the moving support. A "heel" appears at the foot of the curtain. If the heel is sufficiently large, it may contain an eddy in which air bubbles and debris may become trapped, thereby generating a line or streak in the coating. A large heel can also oscillate, producing non-uniformities in the coating along and across the direction of web motion. To prevent puddling, the low viscosity bottom layer may have to be kept thin, even though a functional bottom layer may not be thin, and the curtain height low, though this adversely affects curtain stability and restricts the room beneath the hopper for other equipment, such as a start pan.

It is therefore an object of the present invention to provide a curtain coating process in which high viscosity liquids can be deposited on to a moving support using a low viscosity bottom layer to promote

wetting of the support, but eliminating or significantly mitigating the foregoing disadvantages.

According to one aspect of the present invention, there is provided a curtain coating process in which liquid material comprising two or more layers is coated on to a moving support, and the layer of liquid material adjacent the support has a viscosity which is less than 1mPas.

Advantageously, the bottom layer of low viscosity liquid readily wets the support at high coating speeds and is applied with the curtain as the bottom layer of the liquid material being coated. This allows the liquid material to comprise one or more layers of much higher viscosity, which, in the absence of the bottom layer, would not wet the support so readily and which would, therefore, be more difficult to coat without air-entrainment except at much lower speeds.

The bottom layer may be any low viscosity liquid or solution compatible with the remainder of the liquid material. If the latter comprises aqueous coating compositions, as in the manufacture of photographic products, then the preferred liquid for the bottom layer is water to which small amounts of other substances, such as surfactants, hardeners, dyes etc., may be added as necessary.

By coating a high viscosity curtain with a low viscosity bottom layer, the advantages of high viscosity coating are combined with low viscosity wetting. High coating speeds are possible, and the coating speed at which air-entrainment occurs is less seriously affected by the total flow rate. At the same time, the viscosities of the layers comprising the main bulk of the curtain may be chosen to suit factors other

than wetting such as uniformity, product specification and the drying capacity of the coating track.

According to the present invention, the low viscosity bottom layer may also be very thin (between 0.5 and 10mm). This minimizes the tendency towards puddling and lessens the necessity for the incorporation of non-functional materials. A further advantage of using a thin bottom layer is that diffusion into other layers occurs very rapidly, hence flow after coating is minimized.

In addition, application angles greater than 0° may be used, preferably angles between 20° and 60°. This permits the beneficial use of high curtains whilst again minimizing the tendency towards puddling.

For a better understanding of the present invention, reference will now be made, by way of example only, to the accompanying drawings in which:-

Figure 1 is a schematic cross-section of a curtain coating hopper used in accordance with the present invention;

Figure 2 is schematic partial cross-section of a hopper illustrating an exit slot for a low viscosity bottom layer;

Figure 3 is similar to Figure 2, but illustrates an alternative exit slot arrangement for a low viscosity bottom layer; and

Figure 4 is a schematic cross-section of a curtain coating hopper, but illustrating the use of exit slots as shown in Figures 2 or 3.

A conventional multilayer curtain-coating slide hopper 10 is shown in Figure 1. One method of applying a freely-falling curtain 12 to a moving support 14 is shown, although the support is preferably backed by a precision roller (not shown). The hopper 10 is oriented relative to the moving support 14 so

that the liquid layers flow down the slide surface 28 in a direction opposite to the coating direction. Layer 16 (which wets the support 14), although uppermost on the slide surface 28, becomes the bottom layer of the coating as the curtain 12 impinges on the support 14. The other layers are discharged from respective slots 20, 22, 24 and 26, and are inverted in sequence from that of conventional curtain- or bead-coating.

It is to be noted that the support 14 is inclined to the horizontal so that the angle of application of the curtain to the support, a, is not 0°, that is, the curtain 12 is applied at a forward application angle.

In Figure 2, an exit slot arrangement is shown in which the bottom layer 16 is directly applied to the upstream side of the curtain as it leaves the hopper lip 36. The bottom layer 16 is discharged from an additional slot 30. This slot 30 is formed by inserting a sub-lip element 32 beneath the overhang 34 at the front of the hopper slide 28. In this case, the slot exit is positioned exactly at the hopper lip 36 as shown. However, as shown in Figure 3, the slot 38 can be recessed to form a short slide 40 (of 1cm or less) on the underside of the hopper lip 36. In both these cases, the hopper slide 28 is oriented conventionally in the coating direction as shown in Figure 4 so that the liquid leaving the slots 30, 38 becomes the bottom layer of the liquid material being coated.

In conventional curtain coating processes, the curtain forming hopper is usually oriented in the direction of web travel and the bottom layer is discharged from the bottom slot (i.e. the slot nearest the lip). If this is used for a low viscosity bottom layer such as water, then the coatings are subject to

longitudinal and transverse lines and streaks which are just as unacceptable as those seen in bead coating with the same arrangement. But, by reversing the orientation of the hopper and the order of the other layers, and discharging the bottom layer from the top slot (as discussed with reference to Figure 1), this problem may be removed. In order to achieve this, the hopper slide is inclined at an angle of less than 30° to the horizontal, and the flow rate of the low viscosity liquid layer discharged from the top slot is restricted to not more than one third of the sum of the flow rates of that layer and the high viscosity layer immediately beneath it. Advantageously, the slide surface 16 promotes levelling of any unevenness in the water layer, and the flow on the slide surface is sufficiently stable against waves and other manifestations of slide instability.

A similar advantage is obtained if the bottom layer is applied directly to the upstream side of the curtain (as shown in Figure 4 using the arrangements described with reference to Figures 2 and 3).

In either of the two cases above, if water plus surfactants is used as the bottom layer, uniform coatings can be produced at coating speeds of $600 mmin^{-1}$ or more.

It is possible, therefore, by using curtain processes according to the present invention, to provide uniform multilayer coatings at higher speeds than previously attainable. Although curtain coating is employed, there is flexibility in the choice of flow rate and the viscosities for the layers forming the bulk of the coating. The coating method according to the invention is both product tolerant and easy to engineer with minimum changes in current technology.

Examples of coatings produced using a curtain coating process according to the present invention are now described in the following examples:-

Example 1:

The high viscosity layers comprised an aqueous solution of gelatin having a viscosity of 74mPas at 44°C. At a curtain height of 10.2cm and an application angle of 0°, the maximum coating speed of the gelatin layers alone was typically 324mmin^{-1} at a total wet thickness of 100mm, and 372mmin^{-1} at a thickness of 27mm. Higher coating speeds produced severe air-entrainment.

On addition of a low viscosity bottom layer, 1.1mm in thickness, comprising water containing suitable surfactants, and having a surface tension of 21mNm^{-1} and a viscosity of 0.61mPas at 44°C, uniform coatings were achieved at speeds up to 596mmin^{-1} with gelatin layers having a combined thickness of 45mm. Furthermore, it was found that the thickness of the gelatin layers could be substantially increased by coating at a forward application angle. For example, at an application angle of +20°, uniform coatings of gelatin layers having a combined thickness of 96m were made at speeds of up to 600mmin^{-1} with a low viscosity bottom layer of 4.3mm. In general, uniform coatings were obtainable with low viscosity bottom layers having thicknesses in the range 1 to 8mm, although some successful coatings were made with bottom layers of only 0.5mm.

The main results are summarised in Table 1.

Table 1

Maximum coating speed ($mmin^{-1}$)	Gelatin thickness (μm)	Bottom layer thickness (μm)	Curtain height (cm)	Application angle (°)
324	100	none	10.2	0
372	27	none	10.2	0
596	45	1.1	10.2	0
600	96	4.3	10.2	20

Example 2:

The high viscosity layers comprised an aqueous solution of gelatin having a viscosity of 64mPas at 45°C. At a curtain height of 12.7cm and an application angle of +25°, the maximum coating speed of the gelatin layers alone was typically 376$mmin^{-1}$ at a total wet thickness of 44.3mm. Higher coating speeds produced severe air-entrainment.

Addition of a low viscosity bottom layer, 3.6mm in thickness, having the same composition as that used in Example 1, allowed coatings to be achieved at speeds up to 600$mmin^{-1}$ with gelatin layers having a combined thickness of 96mm. Coating was stable: it was only transiently disturbed by splices and would rapidly re-establish if the curtain was momentarily broken by a rod. However, if the low viscosity layer was interrupted, air-entrainment ensued and it was necessary to slow down and/or reduce flow rates in order to resume uniform coating. Nevertheless, at lower speeds (for example at 465$mmin^{-1}$) uniform coating with heavy laydown (for example a combined thickness of 124mm) would automatically resume following restoration of the supply of the low viscosity bottom layer. With thinner gelatin layers, this reversibility was available at higher speeds.

Results for this example are summarised in Table 2.

Table 2

Maximum coating speed ($mmin^{-1}$)	Gelatin thickness (µm)	Bottom layer thickness (µm)	Curtain height (cm)	Application angle (°)
376	44.3	none	12.7	25
600	96	3.6	12.7	25
465	124	4.6	12.7	25

Example 3:

The high viscosity layers comprised aqueous solutions of gelatin having viscosities of 67.1mPas and 63.2mPas at 42°C. At a curtain height of 25.4cm and an application angle of +45°, the maximum practical coating speed of the gelatin layers alone was typically 313mmin^{-1} at a total wet thickness of 179mm. Higher coating speeds were prone to air-entrainment.

On addition of a low viscosity bottom layer, comprising water containing suitable surfactants, and having a surface tension of 19.3mNm^{-1} and a viscosity of 0.63mPas at 42°C, uniform coatings were achieved at speeds up to 738mmin^{-1} with gelatin layers having a combined thickness of 76mm and a bottom layer thickness of 2.6mm. Coating speeds of up to 600mmin^{-1} were achieved with a bottom layer thickness of 1.1mm and gelatin layers having a total thickness of 93mm.

The results are summarised in Table 3.

Table 3

Maximum coating speed ($mmin^{-1}$)	Gelatin thickness (µm)	Bottom layer thickness (µm)	Curtain height (cm)	Application angle (°)
313	179	none	25.4	45
738	76	2.6	25.4	45
600	93	1.1	25.4	45

Although the examples cited above make use of dilute aqueous surfactant solutions as the low viscosity bottom layer, it is envisioned that the advantages claimed herein could be obtained with a wide range of liquids having viscosities less than 1mPas, such as aqueous solutions of dyes, hardeners and adhesion promoting addenda, and water-miscible and other low viscosity solvents compatible with the other coated layers.

CLAIMS:

1. A curtain coating process in which liquid material comprising two or more layers is coated on to a moving support, and the layer of liquid material adjacent the support has a viscosity which is less than or equal to 1mPas.

2. A process according to claim 1, wherein the layer of liquid material adjacent the support has a wet thickness in a range between 0.5 and 10mm.

3. A process according to claim 2, wherein the wet thickness of the liquid material is in the range between 1 and 5mm.

4. A process according to any one of claims 1 to 3, wherein the liquid material is applied to the moving support at an application angle which is greater than 0°.

5. A process according to claim 4, wherein the application angle lies between +20° and +60°.

6. A process according to any one of the preceding claims, wherein the low viscosity layer is applied as the top layer on a slide surface inclined at an angle of less than 30° to the horizontal, and the flow rate of that top layer comprises not more than one third of the sum of the flow rates of that top layer and the high viscosity layer immediately beneath it.

7. A process according to any one of claims 1 to 5, wherein the low viscosity layer is applied directly to the liquid material from an exit slot positioned at the hopper lip.

8. A process according to any one of claims 1 to 5, wherein the low viscosity layer is applied directly to the liquid material from an exit slot positioned to form a short slide on the underside of the hopper lip.

9. A process according to any one of the preceding claims, wherein the layer of liquid material adjacent the support comprises water including surfactants.

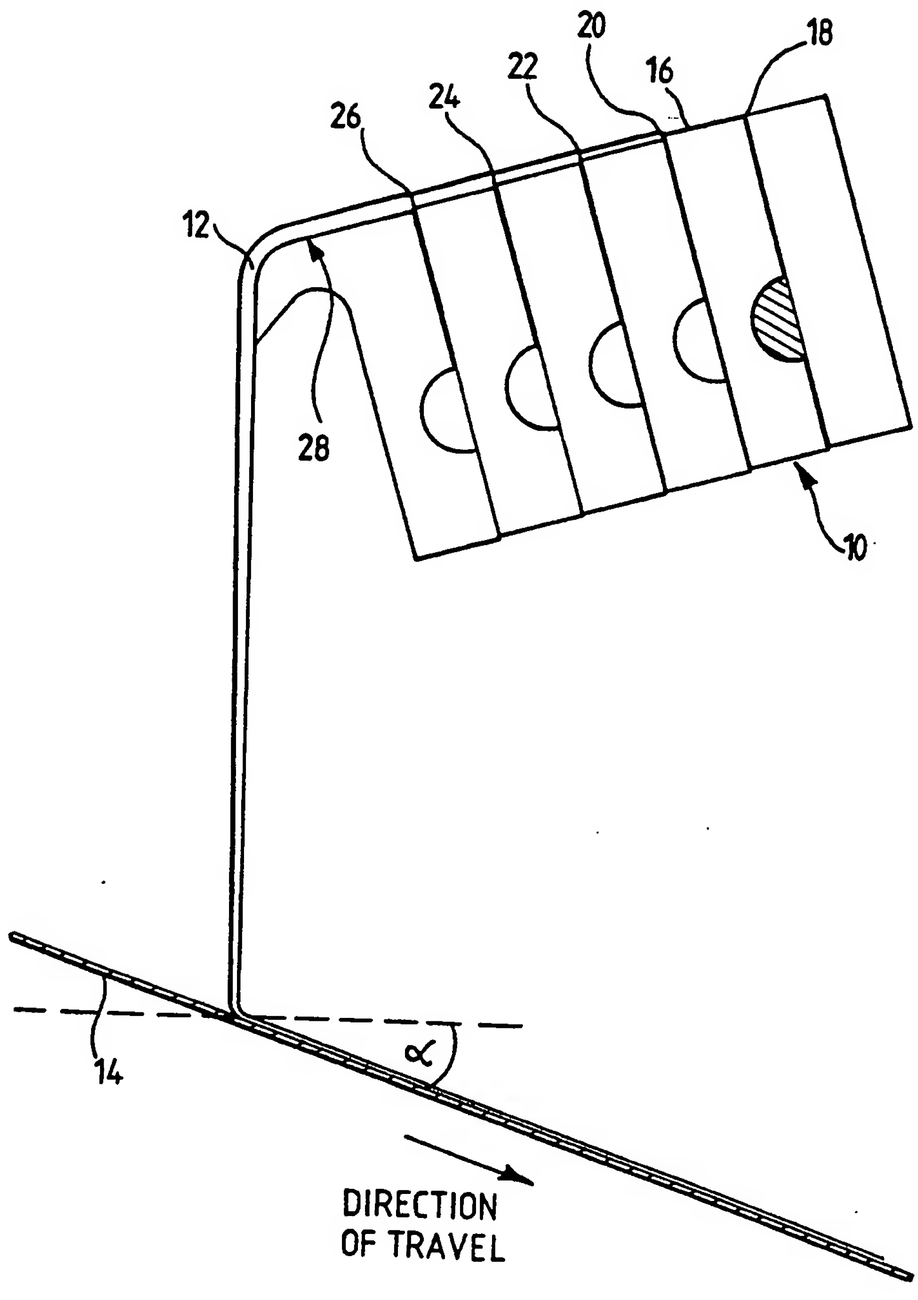

FIG. 1.

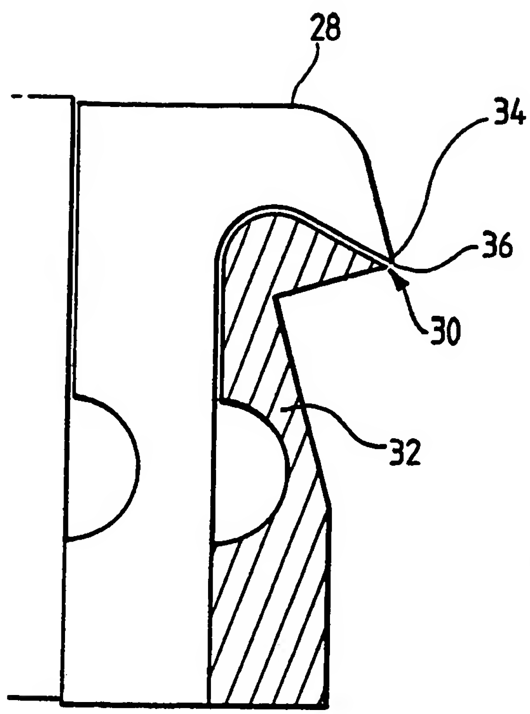

FIG.2.

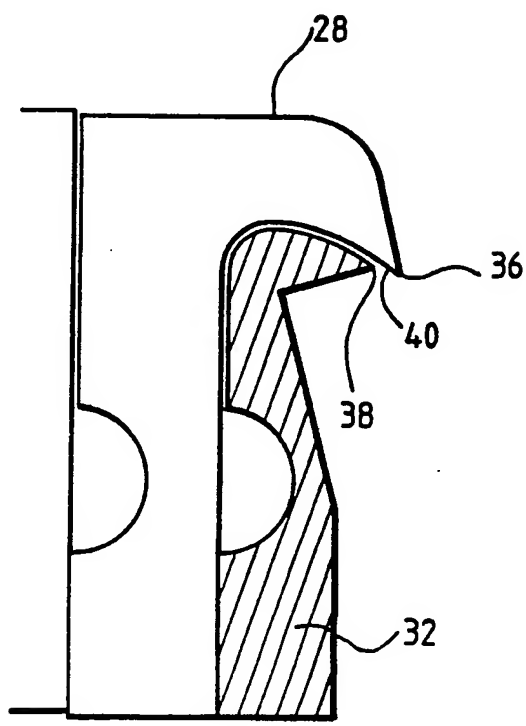

FIG.3.

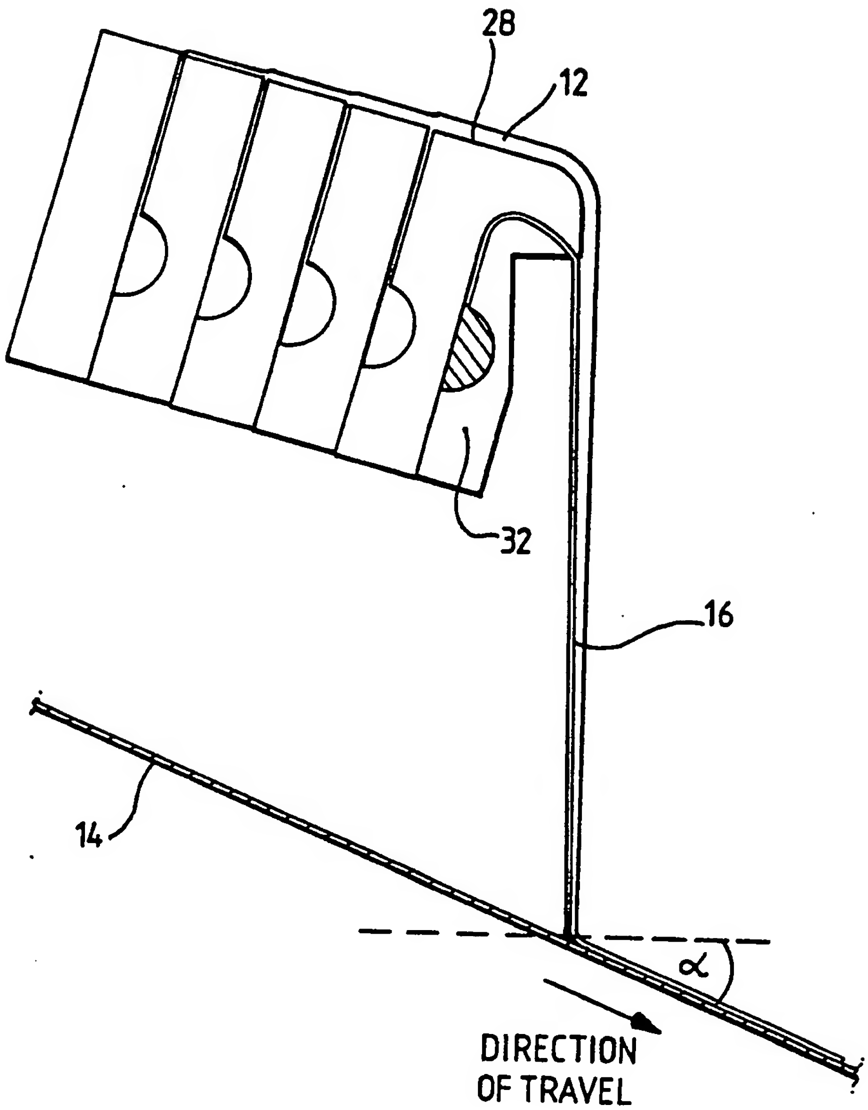

FIG.4.

INTERNATIONAL SEARCH REPORT

International Application No PCT/EP 91/02416

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 B05D1/30; G03C1/74

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	B05D ; D21H ; G03C

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category[°]	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
A	US,A,4 569 893 (AGFA-GEVAERT A.G.) 11 February 1986 see column 3, line 9 - line 18; claims; figures ---	1

° Special categories of cited documents :[10]

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"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
03 MARCH 1992	20.03.92
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	BROTHIER J-A.L.

1

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. EP 9102416 SA 54260

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on 03/03/92
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-4569893	11-02-86	JP-A- 60049343	18-03-85
		JP-A- 60134243	17-07-85
		JP-A- 60134244	17-07-85
		DE-A,C 3431753	21-03-85
		FR-A,B 2551228	01-03-85
		GB-A,B 2148020	22-05-85

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